

## **General Disclaimer**

### **One or more of the Following Statements may affect this Document**

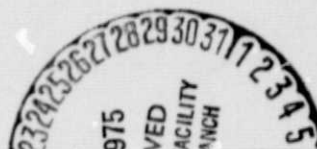
- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

# NASA TECHNICAL MEMORANDUM

NASA TM X-64899

## HIGH-PRESSURE OXYGEN TEST EVALUATIONS

By Robert J. Schwinghamer  
and Carlo F. Key  
Materials and Processes Laboratory



(NASA-TM-X-64899) HIGH-PRESSURE OXYGEN TEST  
EVALUATIONS (NASA) 25 p HC \$3.25 CSCL 21H

N75-31209

Unclas  
35216

G3/20

November 29, 1974

**NASA**

*George C. Marshall Space Flight Center  
Marshall Space Flight Center, Alabama*

1. REPORT NO. NASA TM X-64899		2. GOVERNMENT ACCESSION NO.		3. RECIPIENT'S CATALOG NO.	
4. TITLE AND SUBTITLE  High-Pressure Oxygen Test Evaluations				5. REPORT DATE November 29, 1974	
				6. PERFORMING ORGANIZATION CODE	
7. AUTHOR(S) Robert J. Schwinghamer and Carlo F. Key				8. PERFORMING ORGANIZATION REPORT #	
9. PERFORMING ORGANIZATION NAME AND ADDRESS  George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812				10. WORK UNIT NO.	
				11. CONTRACT OR GRANT NO.	
12. SPONSORING AGENCY NAME AND ADDRESS  National Aeronautics and Space Administration Washington, D. C. 20546				13. TYPE OF REPORT & PERIOD COVERED  Technical Memorandum	
				14. SPONSORING AGENCY CODE	
15. SUPPLEMENTARY NOTES  Prepared by Materials and Processes Laboratory, Science and Engineering					
16. ABSTRACT  The relevance of impact sensitivity testing to the development of the Space Shuttle Main Engine (SSME) is discussed in the light of the special requirements for the SSME. The background and history of the evolution of liquid and gaseous oxygen (LOX/GOX) testing techniques and philosophy is traced from its beginning in the 1950's through the Apollo 13 experience and up to the present. The parameters critical to reliable testing are treated in considerable detail, and the Marshall Space Flight Center (MSFC) 68.94-MN/m <sup>2</sup> (10 000-psia) test apparatus and procedures are described and discussed. Unique design features of the 68.94-MN/m <sup>2</sup> test instrument and their relevance to testing at high pressure are detailed. Discussed also is the high-pressure test rationale, including a contrasting of test requirements at $0.68 \times 10^4$ N/m <sup>2</sup> (1 atm) with those at pressures up to 68.94 MN/m <sup>2</sup> . The test data evaluation is treated at some length, as are the statistical foundations of the actual test practices. Materials threshold sensitivity determination procedures are considered, and a typical MSFC Decision Logic Diagram for sensitivity threshold determination is plotted. Finally, high-pressure materials sensitivity test data are given for selected metallic and nonmetallic materials.					
17. KEY WORDS			18. DISTRIBUTION STATEMENT  Unclassified-unlimited		
19. SECURITY CLASSIF. (of this report)  Unclassified		20. SECURITY CLASSIF. (of this page)  Unclassified		21. NO. OF PAGES  27	
				22. PRICE  NTIS	

## TABLE OF CONTENTS

	Page
INTRODUCTION .....	1
BACKGROUND .....	3
CRITICAL TEST PARAMETERS .....	3
68.94-MN/m <sup>2</sup> TEST APPARATUS AND PROCEDURE .....	4
HIGH-PRESSURE TEST RATIONALE .....	9
IMPACT SENSITIVITY TEST STATISTICS .....	10
IMPACT SENSITIVITY THRESHOLD DETERMINATION .....	12
HIGH-PRESSURE IMPACT SENSITIVITY TEST PRELIMINARY DATA .....	14
Metals .....	14
Nonmetals .....	14
CONCLUSION .....	18
REFERENCES .....	20

# LIST OF ILLUSTRATIONS

Figure	Title	Page
1.	Space Shuttle Main Engine . . . . .	2
2.	Details of striker pin and sample in 68.94-MN/m <sup>2</sup> (10 000-psia) impact sensitivity tester . . . . .	5
3.	68.94-MN/m <sup>2</sup> (10 000-psia) impact sensitivity tester . . . . .	7
4.	Control panel for 68.94-MN/m <sup>2</sup> (10 000-psia) impact sensitivity tester . . . . .	8
5.	MSFC decision logic diagram for impact sensitivity threshold determination . . . . .	13

## LIST OF TABLES

Table	Title	Page
1.	3-Sigma Probability of Getting Number of Reactions Shown for Given Sample Size and Given Percentage of Parent Group Reactions . . . . .	11
2.	High-Pressure LOX/GOX Impact Sensitivity Test Results at 10 kg-m Energy Level — Metals . . . . .	15
3.	Perfluorocarbon Lubricants . . . . .	16
4.	Chlorofluorocarbon Lubricants . . . . .	17
5.	Dry-Film Lubricants . . . . .	18

## HIGH-PRESSURE OXYGEN TEST EVALUATIONS

### INTRODUCTION

For many years the fortunes of space transportation systems have been very dependent upon the understanding, use, and handling of liquid and gaseous oxygen (LOX and GOX). Nor does this crucial relationship show any sign of diminution in the foreseeable future of space transportation activities. The next-generation space transportation system involves a reusable vehicle called the Space Shuttle, so named because of its basic capability of returning at will from earth orbit and being reused many times. The Space Shuttle is being designed to support a wide range of scientific and commercial uses of space such as earth resources work, satellite placement, servicing and repair, materials processing in the absence of gravity, and many other useful tasks. As presently being developed, the vehicle will use a pair of solid-rocket booster motors for liftoff thrust augmentation, while the Orbiter Vehicle itself is to be equipped with three LOX/LH<sub>2</sub> engines, each having a normal sea-level thrust of about 158 757 kg (375 000 lb). The Orbiter engines will operate in the 60 681-MN/m<sup>2</sup> (8800-psia) range.

The practicality of a Space-Shuttle-type vehicle depends upon achieving high efficiencies in propulsion. This type of rocket-propelled vehicle can use to good advantage a vacuum specific impulse (Isp) on the order of 450 to 460 sec, if the liftoff weight and the size of the vehicle are to be minimized. LOX/LH<sub>2</sub>-type engines fulfill this requirement nicely. High-pressure operation of these engines is also very advantageous in such a high-efficiency system. This requires operation at LOX and GOX pressures higher than any used previously in this kind of application. Figure 1 shows a prototype Space Shuttle Main Engine (SSME).

This, then, is the requirement which creates the need to know and understand much better the performance of both metallic and nonmetallic materials in the high-pressure LOX/GOX environment. The Shuttle has been the driving force in the current LOX/GOX test program in extending the Marshall Space Flight Center (MSFC) test capability into the 68.94-MN/m<sup>2</sup> (10 000-psia) test range. A brief perusal of past events will help to put into perspective the chain of events leading to the present LOX/GOX test program.



Figure 1. Space Shuttle Main Engine.



## BACKGROUND

The recorded history of LOX/GOX testing at MSFC dates back to the mid-50's when Lucas and Riehl [1] at the Army Ballistic Missile Agency developed an instrument for acquisition of impact sensitivity data for use by designers then designing the first Saturn space vehicles. The details of this system in the configuration used at that time, called the ABMA Impact Sensitivity Tester, can be found in Reference 2. That same basic tester operating at  $0.68 \times 10^4 \text{ N/m}^2$  (1 atm) but with several modifications is discussed in detail in References 1, 2, and 3 and will not be discussed in any further detail here.

In those early days, an impact sensitivity specification called MSFC-SPEC-106 was also developed which, along with its successors, has been the primary regulatory means during the design of Saturn vehicles. But the crucial happening in NASA which pointed up the urgent requirement for further research on LOX/GOX compatibility at high pressures was the Apollo 13 incident. We refer to the rupture of the No. 2 supercritical oxygen tank in the Command Service Module (CSM) of Apollo 13 about 55 hr after liftoff while en route to the moon. The extensive failure investigations subsequently carried out gave some unprecedented and early insight into the next generation of problems to be solved in the operation of even higher-pressure LOX/GOX systems, such as those now being designed for the Space Shuttle.

Although the pressure in the Apollo 13 failed tank had been about 6206 to 6895 MN/m<sup>2</sup> (900 to 1000 psia), many of the investigators, spurred on by the data they were getting, tested at pressures well beyond those associated with the Apollo CSM operating conditions. This was the case at the MSFC, and the requirement to test at 6895 MN/m<sup>2</sup> (1000 psia) and above provided a headstart on the solution of a whole new generation of problems for the Space Shuttle, which eventually culminated in the development of an impact sensitivity testing system capable of testing to 68.94 MN/m<sup>2</sup> (10 000 psia). Critical test parameters were also surfaced and identified, and the next section discusses some of these parameters in detail.

## CRITICAL TEST PARAMETERS

Investigators at the MSFC have learned through experience with MSFC-SPEC-106B the extreme importance of proper, standardized sample preparation and of cleanliness if reproducible test results are to be obtained. The development of an impact tester to evaluate materials at 68.94 MN/m<sup>2</sup> (10 000 psia)

has reconfirmed conclusively that sample preparation and cleanliness are essential for consistent test evaluations. For example, residual oil or contaminant can easily lead to spurious and extraneous reactions.

Generally speaking, the impact sensitivity of materials usually varies inversely with thickness and directly with pressure [4]. However, factors such as energy rate and energy density delivered to the sample, as well as sample hardness, resilience, and ductility, are also involved in material reactions to impact.

At the MSFC all solid materials are test evaluated in the form of 0.0175-m (11/16-in.)-diameter discs in the specific thickness intended for use in the actual hardware. The 347 stainless steel disc specified by MSFC-SPEC-106B is not used in the high-pressure 68.94 MN/m<sup>2</sup> (10 000 psia) tester because of the necessary deletion of the associated reactive aluminum cups. All materials to be used in high pressure are initially evaluated at 10 kg-m (72 ft-lb) of impact energy at the maximum use pressure and temperature. Materials that indicate evidence of reaction at the standard 10 kg-m energy level are evaluated at decreasing impact energy levels until no reactions are noted in 20 separate test drops at that specific energy level. A new sample is used for each test drop. Any one of the following critical parameters is considered to be evidence of reaction: (1) audible explosion, (2) visible flash, or (3) burning. A test method called the "threshold" determination method is sometimes used which enables the evaluator to obtain the reactivity signature of the material under evaluation. The use of the threshold allows a better comparison of materials and also gives important "rank ordering" information about the relative hazard of specific materials in their use application. This method will be discussed in more detail later.

Specific details of the 68.94-MN/m<sup>2</sup> (10 000-psia) testing system will now be discussed.

## 68.94-MN/m<sup>2</sup> TEST APPARATUS AND PROCEDURE

The tester utilizes the basic ABMA Tester assembly with the addition of a specially designed pressurized sample holder. The tester allows a 9.04-kg (20-lb) plummet to fall through a distance of 1.1 m (43.3 in.). The maximum deviation from free fall allowed is 3 percent. The plummet lands upon the striker pin which protrudes from the sample holder. Figure 2 shows the details and orientation of the striker pin and sample. The basic instrument as

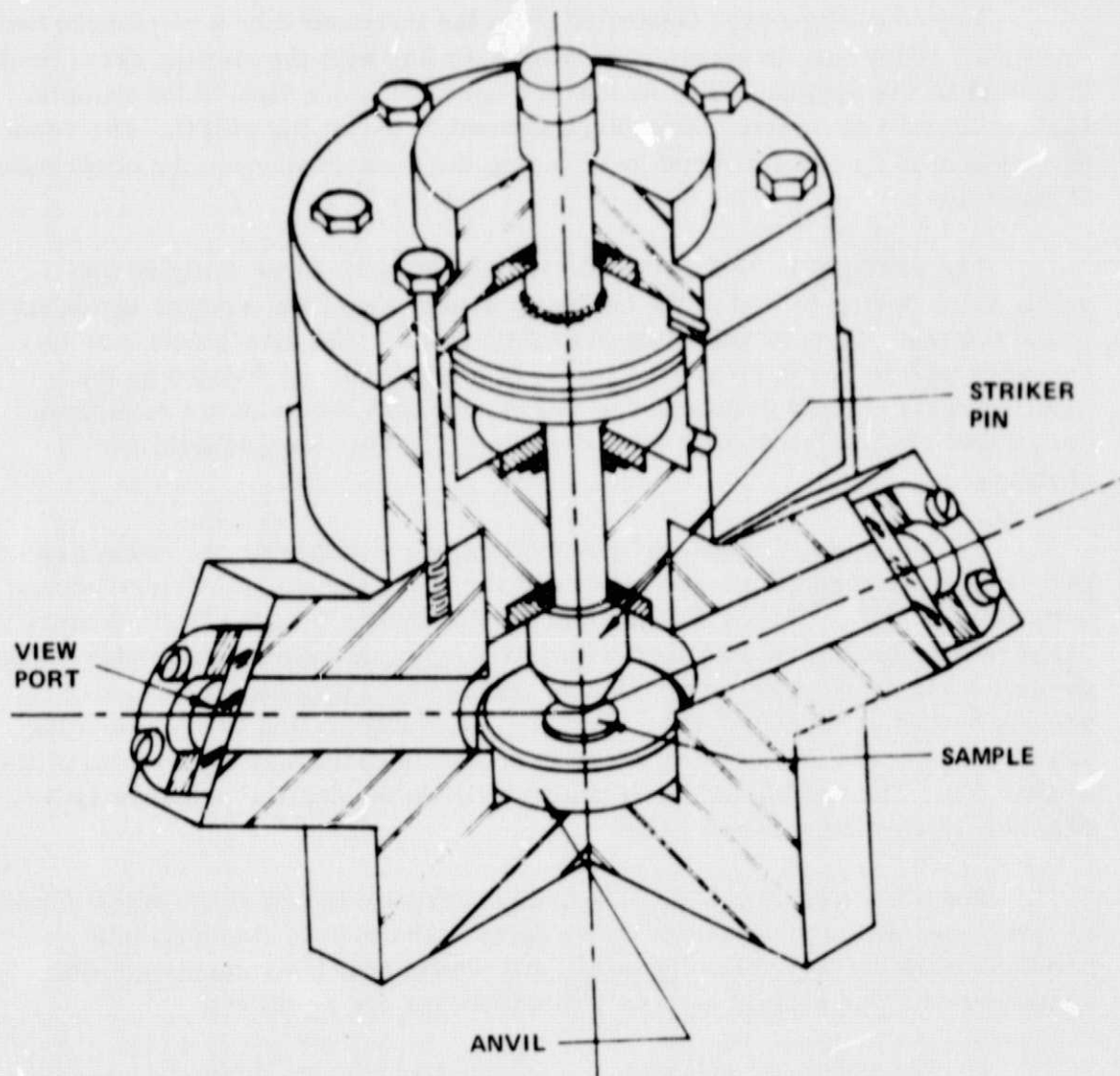


Figure 2. Details of striker pin and sample in  $68.94\text{-MN/m}^2$  (10 000-psi) impact sensitivity tester.

shown consists of a plummet guided in its vertical fall by two sets of bearings, one at each end of the plummet. The bearings arranged at the vertices of equilateral triangles roll freely in steel guide rails. These rails are bolted rigidly to steel tubing supports and are accurately aligned with shims so that uniform contact with the ball bearings is maintained at all points along the length of the rail. The supports are securely anchored to the top and baseplate. The 10.16-cm (4-in.)-thick baseplate is bolted to a rigid metal frame anchored to the concrete floor.

The control panel is separated from the instrument by a reinforced concrete wall containing an observation window in line with the viewing port (flash detector) of the sample holder so that the operator has a view of the sample. Figures 3 and 4 show this testing arrangement in use at the MSFC. The room is darkened to a predetermined level during the tests to enhance the observation of reactions.

The plummet is held at the desired height by an electromagnet and a safety catch spring loaded in the holding position except when power is delivered to the solenoid, thereby releasing the safety catch. The drop height may be varied from 0 to 1.1 m (0 to 43.4 in.). A height indicator located on the electromagnet support strut must be set to zero with the plummet resting on the striker pin and the sample when drop height and/or sample thickness is changed.

The plummet is released by activating two switches on the control panel. One of these releases the safety catch and the other reverses the electromagnetic field, which releases the plummet as the field collapses. The plummet delivers the impact to a stainless-steel striker pin resting on the sample. The position striker assembly is of a unique design which provides a 12.4-to-1 pressure ratio to "balance" the system and precludes having to overcome the full 68.94 MN/m<sup>2</sup> (10 000 psia) working against the cross-sectional area of the striker pin. The sample holder is pressurized to the desired pressure by a remotely controlled solenoid valve.

The high-pressure LOX/GOX tester currently in use at the MSFC (built to MSFC specified requirements by Rocketdyne Division of Rockwell International) contains increased diagnostic and operational instrumentation which compared with our earlier system. The additions are as follows:

1. The striker is balanced at any pressure with an automatic pressure-balancing system.
2. All high-pressure operations are remote and pressures above 1397 MN/m<sup>2</sup> (2000 psig) are created by a two-stage diaphragm compressor.
3. Additional automatic and logic control circuits are used in the graphic control panel for convenience, reliability, and safety of operation.
4. A four-channel, dual-beam oscilloscope is an integral part of the instrumentation readout.

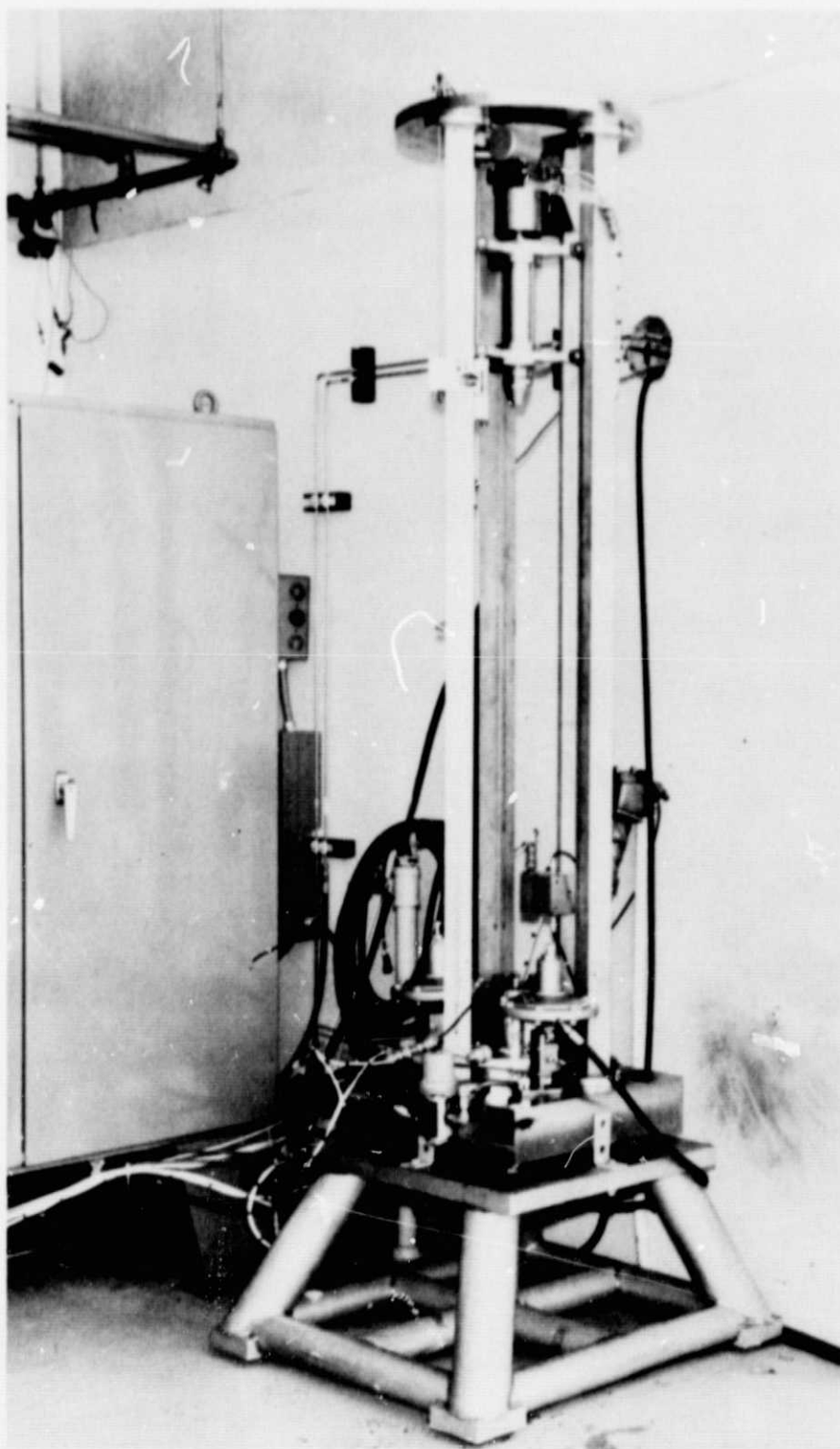


Figure 3.  $68.94\text{-MN/m}^2$  (10 000-psia) impact sensitivity tester.

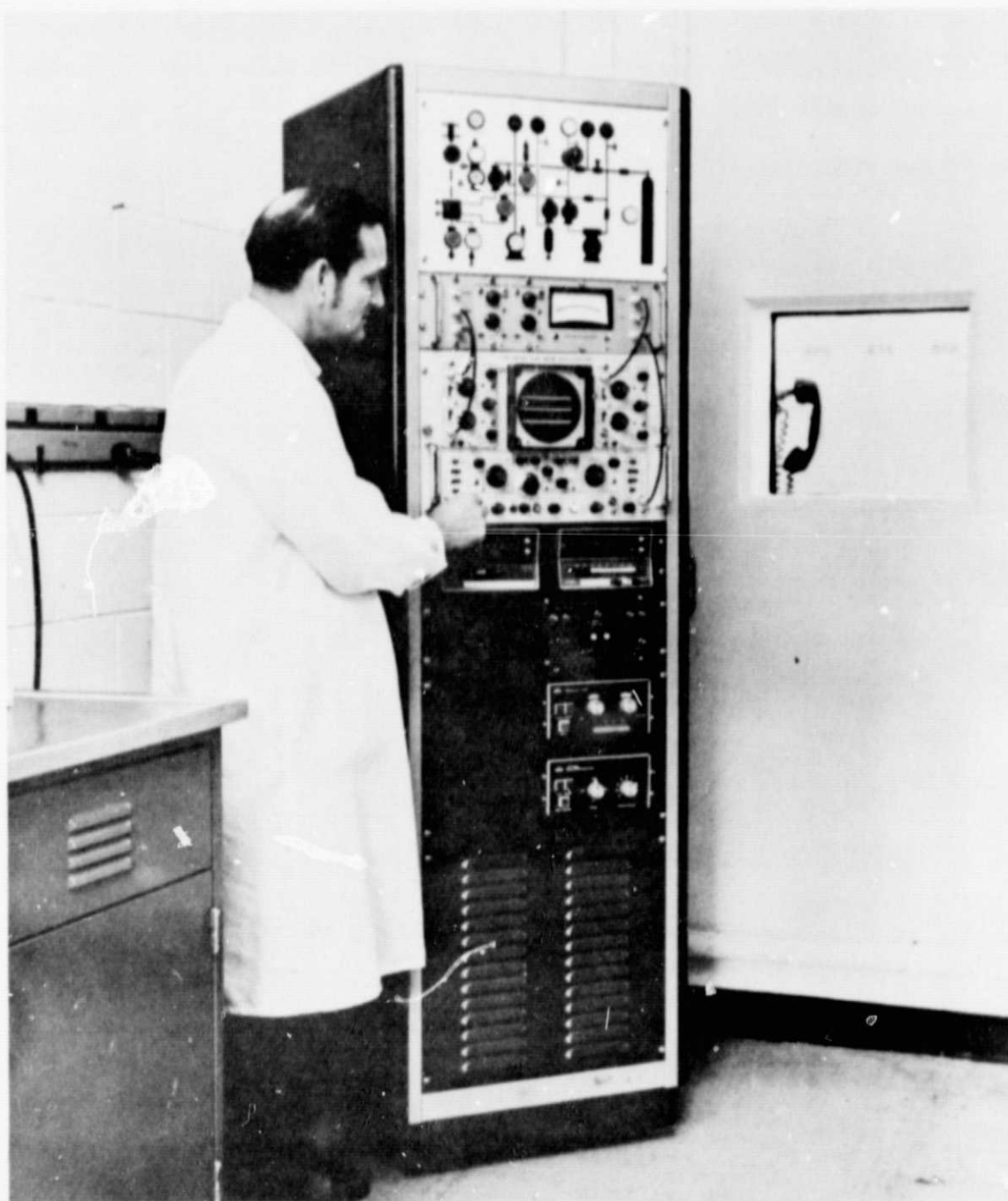


Figure 4. Control panel for  $68.94\text{-MN/m}^2$  (10 000-psia) impact sensitivity tester.

5. A camera attached to the scope allows the recording of four variables at impact: plummet velocity, photocell flash, high-frequency pressure response, and load-cell response.

6. The load-cell response is an actual analog display of the energy transferred to the sample.

7. Sample temperature and cell pressure are continuously monitored by digital readout meters.

Two other basic design features of the high-pressure tester should be reiterated: the use of the balanced piston and a rebound catcher. As noted above, the balanced piston concept was chosen to eliminate pressure effects on the system, thereby decreasing friction and assuring that the striker pin is always in contact with the sample. The pressure used to balance the piston is automatically controlled to ensure both minimum drag and intimate contact of the striker pin with the sample. A rebound catcher is used to eliminate rebound reactions and thereby eliminate one potential variable. Unlike the basic ABMA Tester, the more sophisticated diagnostics used in this system enable the operator to determine more precisely the energy delivered to the sample. Because of detailed analysis of many oscilloscope traces of rebound response, it was decided to employ a catcher during testing of materials at these high pressures.

## HIGH-PRESSURE TEST RATIONALE

The actual selection of the number of drops or impacts (sample size) to use to obtain good statistically meaningful data has been a subject of debate since the very beginning of LOX impact sensitivity testing. As noted earlier, the system used at the MSFC consists of a basic sample size of 20 drops. If no reactions result in these drops, then the material is passed. If 1 reaction in 20 drops occurs, then 40 additional consecutive drops without any more reactions must be successfully accomplished for the material to pass unconditionally. This system has been used successfully for 15 years now in testing at  $0.68 \times 10^4 \text{ N/m}^2$  (1 atm) and it has covered about 2000 different materials with a total number of drops of about a quarter million. On new materials of largely unknown composition, frequently as many as 300 drops will be made on a given material to ensure an even more statistically substantiated result. No failure has occurred in any LOX system attributable to impact sensitivity in the 15 years since the inception of this test.

Unfortunately, in the high-pressure testing regime, two factors preclude the arbitrary imposition of the  $0.68 \times 10^4 \text{ N/m}^2$  (1-atm) test criteria:

1. Because of the high-energy density used in the  $0.68 \times 10^4 \text{ N/m}^2$  (1 atm) test, many materials we need to use simply cannot pass the test at the highest pressures now of interest; this is especially true of nonmetallics.
2. The inordinate amount of time required to test at high pressure, higher temperatures, or both, precludes several hundred drops on any given material at the use pressure and temperature conditions.

At pressures of  $68.94 \text{ MN/m}^2$  (10 000 psia), it is indeed difficult to find nonmetallic materials capable of passing the impact sensitivity test with the standard  $10\,185\,872 \text{ kg/m}^3$  (388 lbm/in.<sup>3</sup>) energy density delivered to the sample at a 10 kg-m (72 ft-lb) total drop energy level. However, it is recognized and intended that the 10 kg-m energy level constitute a stringent test of the material. Initially, this level was set to provide some margin of safety to compensate to some extent for the impracticality involved in making many hundreds, or thousands, of drops which naturally would provide a higher statistical assurance.

## IMPACT SENSITIVITY TEST STATISTICS

To plead the case for a high-impact energy test level to offset the statistical disadvantage of fewer drops, a look at the basic applicable statistics is in order. If, therefore, one wishes to determine statistically the probability of getting 0 reactions in 20 drops when the parent group (the population or the "production" material) is running to about 10- to 49-percent reactions, the Binomial Test can be successfully applied [5]. If the parent groups, or the "production" material, is running less than 10-percent defectives (getting reactions less than 10 percent of the time), then the Poisson's Test is sometimes better for predicting the probability of 0 reactions in 20 drops. These statistical criteria constitute the basis for the chart shown in Table 1. This table gives the 3-sigma (99.7-percent) probability of getting the number of reactions shown in the body of the chart, for a given sample size and a given percentage of reactions in the population (production material). It is immediately apparent that zero reactions in 20 drops become significant only if the material is running typically on the order of 30-percent reactions. It can also be concluded that there is a 99.7-percent probability of getting from 1 to 12 reactions in a given series of 20 drops, on that same material, if it has a 30-percent reaction rate.



TABLE 1. 3-SIGMA PROBABILITY OF GETTING NUMBER OF REACTIONS SHOWN FOR GIVEN  
SAMPLE SIZE AND GIVEN PERCENTAGE OF PARENT GROUP REACTIONS

Sample Size	Typical Reactions in the "Population," or Production Material (%)						
	1	5	10	20	30	40	50
4	0-1	0-2	0-2	0-2	0-4	0-4	0-4
9	0-1	0-2	0-4	0-5	0-7	0-8	0-9
16	0-2	0-3	0-5	0-8	0-10	0-12	2-18
20	0-2	0-4	0-7	0-10	1-12	2-14	3-16
25	0-2	0-5	0-7	0-11	1-14	3-17	5-20
36	0-2	0-6	0-9	0-14	3-19	6-23	9-27
49	0-3	0-7	0-11	1-18	5-24	9-30	14-35
64	0-3	0-8	0-14	3-22	8-30	14-37	20-44
81	0-3	0-10	0-16	5-27	12-37	19-45	27-54
100	0-4	0-12	1-19	8-32	16-44	25-55	35-65
121	0-4	0-13	2-22	11-37	21-52	32-65	44-77
144	0-5	0-15	4-25	14-43	27-60	40-75	54-90
169	0-5	0-17	5-29	18-49	33-69	49-87	65-104
196	0-6	0-19	7-23	22-56	40-78	58-99	77-119
225	0-7	1-21	9-36	27-63	47-88	68-112	90-135
256	0-7	2-23	11-40	32-70	55-99	79-126	104-152
400	0-10	7-33	22-58	57-103	92-143	131-189	170-230
900	2-18	25-65	63-117	144-216	229-311	317-403	405-495

These statistical facts indicate that whenever possible the number of drops (samples) should be as large as any given program schedule will allow. But, in the real world of LOX/GOX testing, a completely and totally statistically adequate number of samples can never be taken. For this reason, a satisfactorily high level of impact energy must be used so that, in addition to the statistical assurance of rejecting materials in the 10-percent reactions and above range, we also have the assurance that the test energy level is significantly higher than any flight hardware design will ever incorporate. The following are the two sturdy legs upon which stands the colossus of test data built thus far in the MSFC LOX/GOX impact sensitivity test program:

1. Enough samples to screen effectively to at least 10-percent reactions level.

2. Test energy level higher than any hardware design uses. High-pressure LOX/GOX testing now, however, stands at a critical juncture. Preliminary data indicate that only very few nonmetallics can be used in gaseous oxygen in systems delivering energy densities comparable to the standard 10 kg-m (72 ft-lb) test magnitude. Therefore, if the standard test level is too high, what must inevitably follow is a comparison of the actual hardware system energy to be expected versus the specific material threshold energy level. We call this the "threshold determination." This method requires a critical assessment of the functional parts of the hardware including adiabatic compression effects, kinetic energy of striking or sliding parts, and other momentum and impact effects. One compares the hardware available energy with the sensitivity threshold as determined by the test procedure, and the margin of safety must then be considered.

## IMPACT SENSITIVITY THRESHOLD DETERMINATION

Figure 5 shows the MSFC decision logic diagram for determination of the impact sensitivity threshold. The sequence of events proceeds from left to right. The test begins with 20 drops, with one of three possible change events resulting. If the material has 0 reactions in 20 drops, the material passes unconditionally. One reaction in 20 drops brings up decision 2b, at which point acceptance of the material is made contingent upon 40 more consecutive drops without any further reactions. We are now proceeding down the center branch of the "decision tree," from left to right. Two chance events can occur at this point: 1 in 60 (pass) or 1 in 60, in which case this branch of the "tree" brings decision 3c, which is either to fail the material at 10 kg-m (72 ft-lb) or to



"go for threshold," beginning by an incremental drop to 9.1 kg-m (65 ft-lb). This procedure is repeated, each time lowering the impact energy incrementally until 0 in 20 or 1 in 60 drops can be substantiated. This, then, is the impact sensitivity threshold for that material. This value is then the criterion against which the actual hardware design must be assessed.

Going back to the left of the diagram in Figure 5 again, the top branch of the first chance event can be followed through in a similar manner, the only difference being that unconditional failure at 10 kg-m (72 ft-lb) is evident earlier than in the center branch. While at first glance the logic diagram may appear somewhat complex, it does constitute an orderly, logical, and methodical approach to a complex situation and in actual practice eliminates much subjectivity in conducting the test operation.

## HIGH-PRESSURE IMPACT SENSITIVITY TEST PRELIMINARY DATA

### Metals

The test evaluations of many of the metals considered for use in the SSME are given in Table 2. The nickel base superalloys Rene' 41, Inco 625, and Inco 718; the nickel-copper alloys, Narloy X and K Monel; and the cobalt base superalloy HS 188 all passed the impact sensitivity test at the test pressure of 68.94-MN/m<sup>2</sup> (10 000 psia) and the use temperature. The high-strength aluminum casting alloy Tens-50 showed no reactions in 20 trials at 68.94 MN/m<sup>2</sup> at LOX temperature but exhibited 1-percent reactions (1/2) in 2 trials at 68.94 MN/m<sup>2</sup> and 300°K (81° F) in GOX. This was not wholly unexpected since the reactivity in GOX of aluminum denuded of its protective oxide coating is well known, whereas on the other hand, the enormous beneficial thermal inertia and consequent quenching effects on aluminum in LOX have been observed many times in impact sensitivity testing. This same beneficial heat-sink effect can also be observed with nonmetallics as well. Large quantities of Inco 718 are used in the SSME design, and the test data show this material to be an excellent choice because of its superior resistance to reaction in LOX and GOX.

### Nonmetals

Lubricants (fluid). A number of lubricant materials which had previously met the MSFC-SPEC-106B criteria [basic test pressure,  $0.68 \times 10^4$  N/m<sup>2</sup> (1 atm)] have been evaluated at various pressures up to 10.3 MN/m<sup>2</sup> (1500 psia). These materials were basically a variety of perfluorocarbon,

TABLE 2. HIGH-PRESSURE LOX/GOX IMPACT SENSITIVITY  
TEST RESULTS AT 10 kg-m ENERGY LEVEL — METALS

Material	Material Thickness [cm (in.)]	Test Temperature [° K (° F)]	Reactions (%)
Aluminum Tens-50	0.127 (0.050)	90 (-297)	0
	0.127 (0.050)	300 (81)	50 <sup>a</sup>
HS 188	0.031 (0.012)	420 (+296)	0
Inco 625	0.127 (0.050)	418 (+293)	0
Inco 718	0.031 (0.012)	300 (81)	0
	0.127 (0.050)	300 (81)	0
Ag Plated Inco 718	0.160 (0.063)	300 (81)	0
	0.160 (0.063)	355 (47)	0
K Monel	0.127 (0.050)	300 (81)	0
Narloy X	0.127 (0.050)	90 (-297)	0
Rene' 41	0.135 (0.053)	358 (185)	0

a. Sustained burning.

chlorofluorocarbon and inorganic bonded dry film lubricants. The perfluorocarbon lubricants (Krytox, Brayco, and Fromblin Series Z) revealed no evidence of reactivity at the test pressures and temperatures used, as shown in Table 3.

The chlorofluorocarbon lubricants (consisting of Halocarbons plus Fluorolubes) did show an increase in reactivity in the case of three fluids, Halocarbon 10-25ES, Halocarbon 14-25 and Fluorolube GR-290, in the test medium of GOX at 10.3 MN/m<sup>2</sup> (1500 psia). The data on Table 4 show a 40-percent reactivity in 10.3-MN/m<sup>2</sup> GOX at 10-kg-m (72-ft-lb) energy level for Fluorolube GR-290 and a 60-percent reactivity in 10.3-MN/m<sup>2</sup> GOX at a 10-kg-m energy level for Halocarbon 10-25ES. On the other hand, Halocarbon 14-25 exhibited only a 10-percent reactivity under similar conditions. Additionally, a second batch of Halocarbon 10-25ES was tested (see Table 4, No. 2 item under Halocarbon 10-25ES), and it exhibited no reactions when tested to the same conditions as the No. 1 batch. This is a good example of the problem




TABLE 3. PERFLUOROCARBON LUBRICANTS

Material	Thickness [cm (in.)]	Pressure [MN/m <sup>2</sup> (psia)]
Brayco-813	0.0762 (0.030) ↓	0.68 (100)
		3.4 (500)
		6.8 (1000)
		10.3 (1500)
Brayco-810		0.68 (100)
		3.4 (500)
		6.8 (1000)
		10.3 (1500)
Brayco-812		0.68 (100)
		3.4 (500)
		6.8 (1000)
		10.3 (1500)
Fromblin Fluid Series Z		10.3 (1500)
Krytox 240AC		10.3 (1500)
Krytox 290AB		0.68 (100)
		3.4 (500)
		6.8 (1000)
		10.3 (1500)

confronting the LOX/GOX sensitivity tester. Is this evident disparity due to (1) statistical probability, (2) batch sensitivity, or (3) sample contamination? This is a case in which there is no substitute for further testing and, indeed, this is the only way the answer can be found.

In any case, the ancillary point needs to be made here that the general idea that chlorofluorocarbon lubricants are always acceptable for use in LOX/GOX systems needs to be viewed with a degree of skepticism. Further testing should disclose the reason for the disparity noted above, but in the interim all batches of this material should be evaluated for impact sensitivity at the use pressure and temperature.

TABLE 4. CHLOROF LUOROCARBON LUBRICANTS

Material	Thickness [cm (in.)]	Pressure [MN/m <sup>2</sup> (psia)]	Energy Level (kg-m)	Reaction (%)	
				LOX	GOX
Fluorolube LG-160	0.0762 (0.030)	10.3 (1500)	10	0	0
Fluorolube GR-290		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	40
		10.3 (1500)	9	-	25
		10.3 (1500)	8.3	-	0
		10.3 (1500)	7.6	-	0
Halocarbon 4-11ES		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	0
Halocarbon 10-25ES		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
	10.3 (1500)	10	0	60	
	10.3 (1500)	8.3	-	10	
	10.3 (1500)	7.5	-	0	
	10.3 (1500)	7.0	-	0	
Halocarbon 14-25	0.68 (100)	10	0	0	
	3.4 (500)	10	0	0	
	6.8 (1000)	10	0	0	
	10.3 (1500)	10	0	0	
		9	-	10	
		8.3	-	0	
Halocarbon 14-25ES	0.68 (100)	10	0	0	
	3.4 (500)	10	0	0	
	6.8 (1000)	10	0	0	
	10.3 (1500)	10	0	0	

Lubricants (dry film). Several inorganic, bonded, dry-film-type lubricants which generally meet the MSFC-SPEC-106B criteria have also been evaluated at  $10.3 \text{ MN/m}^2$  (1500 psia) in both LOX and GOX. Table 5 shows the results of these evaluations. Here again, materials of ostensibly the same basic composition react differently to the same test conditions, and, again, it is evident that further testing is mandatory to establish unequivocally the precise differences. By referring to Table 1, one can easily infer how such apparently ambiguous results can occur. The table shows that zero reactions would be meaningful only if the number of reactions in the parent group were running as high as 30 percent. Equally obviously, increasing the sample size to a few hundred will very quickly allow one to "home in" on the real reason for the apparent ambiguity.

Equally important, the fact that such an ambiguity exists should not be grounded for any adverse criticism relative to either the lubricants or the manufacturer. Further testing will disclose the reasons, whether they are statistically or batch-sensitivity related. These ambiguous data are shown not to call attention to the statistical inadequacies of any given test method but to reiterate the tremendous benefit to be gained by using test energy levels well above any of the worst-case levels to be expected in the actual hardware application and to point out the great desirability of using the very largest sample sizes that time and money considerations will allow.

## CONCLUSION

In conclusion, we want to reemphasize the great statistical and practical importance of having the largest possible test sample size consistent with cost and time considerations, and above all, the enormous advantage of using test energy density levels considerably above anything the actual hardware can be expected to produce, even under worst conditions. With these thoughts in mind, we are working on the new-generation, high-pressure LOX/GOX materials sensitivity problems with optimism and confidence.



TABLE 5. DRY-FILM LUBRICANTS

Material	Thickness [cm (in.)]	Pressure [MN/m <sup>2</sup> (psia)]	Energy Level (kg-m)	Reaction (%)	
				LOX	GOX
Electrofilm 2306	0.0102 (0.004) ↓	1 atm (14)	10	0	0
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	10
		6.8 (1000)	9	-	0
			8.3	-	0
		10.3 (1500)	10	0	20
			9	-	0
			8.3	-	0
Electrofilm 2396	↓	1 atm (14)	10	0	
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	30
		10.3 (1500)	9	-	25
			8.3	-	15
			7.6	-	0
			6.9	-	0
Electrofilm 2406	↓	1 atm (14)	10	0	-
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	0
Everlube 811-B	↓	1 atm (14)	10	0	-
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	0
Everlube 812	↓	1 atm (14)	10	0	
		0.68 (100)	10	0	0
		3.4 (500)	10	0	10
		6.8 (1000)	10	0	30
		10.3 (1500)	10	0	40
Inlox 44	↓	1 atm (14)	10	100	-
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	0
Inlox 88	↓	1 atm (14)	10	0	-
		0.68 (100)	10	0	0
		3.4 (500)	10	0	0
		6.8 (1000)	10	0	0
		10.3 (1500)	10	0	30
	-	-	9	0	30
	-	-	8.3	-	15
	-	-	7.62	-	0
	-	-	6.95	-	0

## REFERENCES

1. Lucas, W. R.; and Riehl, W. A.: An Instrument for the Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen. ASTM Bulletin No. 244, February 1960, pp. 29-34.
2. Curry, J. E.; and Riehl, W. A.: Compatibility of Engineering Materials with Liquid Oxygen. Report MTP-M-S&E-M-61-7, George C. Marshall Space Flight Center, Huntsville, Ala., March 21, 1961.
3. Key, C. F.: Compatibility of Materials with Liquid Oxygen. NASA TM X-64711, Oct. 1, 1972.
4. Schwinghamer, R. J.: Impact Sensitivity of Materials in Contact with Liquid and Gaseous Oxygen at High Pressure. Paper presented at Symposium on Oxygen Compressors and Pumps, Compressed Gas Association, Inc., Atlanta, Nov. 9-11, 1971.
5. Langley, Russell: Practical Statistics. Dover Publications, Inc., New York, 1970.

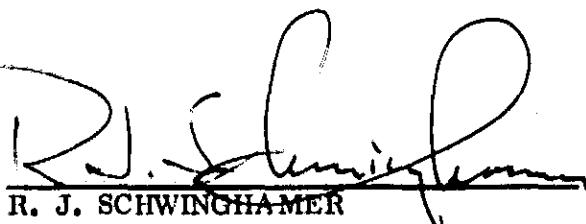
## APPROVAL

### HIGH-PRESSURE OXYGEN TEST EVALUATIONS

By Robert J. Schwinghamer and Carlo F. Key

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

A handwritten signature in black ink, appearing to read 'R. J. Schwinghamer', is written over a horizontal line.

R. J. SCHWINGHAMER  
Director, Materials and Processes Laboratory